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Abstract

When cementitious materials are used for the containment of waste they act in two different, and sometimes conflicting, ways. The first is physical containment in which the waste is physically isolated from the environment. The second is chemical containment in which the water passing through the barrier is buffered to high pH thereby substantially reducing the solubility of many harmful species and promoting sorption onto the matrix. Chemical barriers have been extensively researched for nuclear waste containment and this paper reports on the transfer of technology developed for the underground storage of nuclear waste to the area of non-nuclear waste management. It focuses on the ability of a cementitious barrier to chemically condition liquid leachates, neutralising organic acids and reducing the solubility of priority pollutants such as transition metal ions and arsenic. This is similar to the way in which cement suppresses actinide leaching in the disposal of nuclear wastes.

Recent increases in disposal costs including the introduction of the landfill tax have made low cost low strength mixes a financially attractive option as a barrier for landfill leachate containment. In this paper the results of an extensive preliminary investigation into potential mixes using various mineral wastes are presented and some of the measured properties are compared with those, which are required for a multi-layer barrier. The cementitious mixes, which have been proposed, contain large amounts of secondary materials. If these materials were treated as wastes their disposal costs would be high, so the mixes may be designated "negative cost mixes". The results indicate that many of these mixes are well suited to this application.

Keywords: cementitious mixes; cementitious chemical buffering; mineral wastes.

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1. Introduction

Current landfill liner practice relies largely on the use of polymer membranes protected by sand or a sand-bentonite overpack. These protect the geosphere from the release of leachates. Typically, such membranes are used below a geotextile layer and a drainage blanket, allowing leachate to collect at a sump, prior to pumping and treatment. This construction is both expensive and vulnerable to puncture during its emplacement and susceptible to rupture during the operational phase of the landfill.

To protect the liner systems during waste emplacement, waste must be sorted to allow a layer of graded waste to protect the liner from sharp objects which may be present in the bulk of the waste. Waste sorting and emplacement of this so called 'fluff' layer of graded waste, adds to the operators' costs and leaves waste vulnerable to wind dispersal.

The vast majority of commercial applications of (and hence research efforts into) landfill liners focuses on HDPE based systems, yet interest in mineral barriers continues to grow, especially overseas [1]. Current research is concentrated in three subject areas: geotechnical investigations into the composite sand-clay-geotextile system, leak detection studies undertaken on behalf of both the regulators and operators [2] and research into waste leaching [3 – 5]. The latter has resulted in the formation of an EC thematic network on leach testing procedures [6]. Similar work in construction materials has generated a wealth of knowledge in the leaching of cements and concretes, which has also resulted in an EC thematic network being established [7]. The CEN have drawn on these initiatives along with those of the national standards authorities in Europe and North America [8] to produce draft standard procedures for waste characterisation testing of wastes and construction materials.

Michell and van Court [9] and Rumer and Ryan [10] provide comprehensive coverage of most details of containment barrier systems. The major function of all barrier systems is to provide isolation of wastes from the surrounding environment, thereby protecting groundwater, soil, and air from contamination. Thus, the ability of any barrier to prevent movement of waste liquids and gases through it is of paramount importance. Accordingly, the hydraulic and gas conductivities of properly manufactured and installed barrier materials are the properties of greatest concern. In the Europe and U.S.A it is usually required that the hydraulic conductivity (coefficient of permeability i.e. k defined from Darcy velocity $V = k (h_1-h_2) / x$, (m/s) where the fluid is flowing through a thickness x (m) with pressure heads h_1 and h_2 (m) on each side) be less than 10^{-9} m/s for earth and treated earth barrier materials, with values up to 10^{-8} m/s allowed in some cases [11]. Geomembrane barriers are usually

specified in terms of minimum acceptable thickness and material type rather than a maximum allowable conductivity.

The use of soil cement as a containment barrier for toxic waste has focused on double barriers, following U.S. guidelines. This involved the sandwiching of an impermeable flexible membrane between two layers of soil cement. Initial results have proved encouraging. Dinchak[12], Schevon and Damas[13], Anon[14].

The cementitious chemical barrier is one of the main engineering features of the current plans for a UK repository for medium and low level nuclear waste. The concept has been developed in response to a requirement for a barrier, which will have a predictable performance in a deep saturated geological environment over a timescale of up to a million years. The barrier is built out of conventional engineering materials but its method of operation is far from conventional for an engineering structure because it is essentially sacrificial. The main function of the barrier is to condition the chemistry of the repository to high pH by dissolving alkalis in the groundwater. The alkalis are free sodium, potassium and calcium and subsequently the calcium silicate hydrate which forms the structure of the hardened cement.

Physical containment with concrete is well understood and documented [1, 15 & 16]. The degree of containment will depend on the permeability of the barrier. The permeability of concrete and mortar are relatively easy to measure and this has been achieved in a wide variety of different ways. One of the better methods is the use of a Hoek cell [17].

Chemical containment has been studied in detail for nuclear waste [18]. In the type of repository for which a chemical barrier would be used the main mechanism of loss of radionuclides is caused by flowing groundwater. This flow may be present in the area before the repository is built or it may be caused by the heat generated in the repository. In order to operate for a long time a chemical barrier depends on other barriers to limit the flow of groundwater through it. This is normally achieved by positioning the repository in a geology with a very low permeability. In this situation the permeability of the repository itself can be shown not to have a significant effect on the flow of water through it.

Thus water will enter the repository very slowly and the chemical barrier works by conditioning it before it reaches the waste and also after it leaves the waste but before it leaves the repository. Before it reaches the waste the barrier will raise the pH of the water, reduce the Eh, and remove many dissolved ions such as sulphates. In this way the barrier will ensure that the solubility of the radionuclides in the waste is as low as possible. For example raising the pH from 8 to 12.5 will reduce the solubility of

Uranium by an order of magnitude, Plutonium and Protactinium by one and a half and Americium by three and a half. After the water leaves the waste the barrier will provide a high capacity for sorption to remove radionuclides from it.

It may be seen that, unlike a conventional engineering structure, the method of operation of a chemical barrier is sacrificial. As it operates the cement matrix carbonates and reacts with sulphates and other materials to an extent, which would indicate failure in a conventional structure.

The authors are currently working on a major industry-based project on a novel composite barrier system, which uses the materials described in this paper and is shown in figure 1 [19]. This work includes carrying out large-scale site trials to demonstrate the construction of the system. The trials consist of cells approximately 8m wide, which are designed to contain leachate to a depth of 1m maximum allowable leachate level in current landfill practice and are made with the candidate barriers [20]. In this paper the results of an extensive preliminary investigation into potential mixes using various mineral wastes are presented and some of the measured properties are compared with those, which are required for a multi-layer barrier. The cementitious mixes, which have been proposed, contain large amounts of secondary materials. The results indicate that many of these mixes are well suited to this application.

2. Design philosophy of composite barriers

The novel multi layer barrier concept is based on the theory that the pollution of soils and watercourses by the release of leachate may be prevented by adoption of a composite-barrier liner, which not only chemically conditions the waste, but is designed to be self-sealing through secondary mineralisation and will retain heavy metal ions through ion exchange, surface sorption, filtration and precipitation.

The requirements of a landfill liner (barrier) are two-fold: it must be physically strong enough to allow vehicular access during the operational phase, and provide adequate containment of leachate during the post-closure period. In order to satisfy both these operational and long-term requirements, a range of composite barrier materials have been evaluated. These include: low cost, chemically conditioning, cementitious media (*e.g.* concretes containing limestone, metallurgical slags, spent foundry sands and/or demolition waste as an aggregate; blended cements containing waste materials such as fly ash and slag) and non-swelling clays.

The properties of an ideal barrier system are:

- Low permeability. This must be less than 10^{-8} ms^{-1}.
- High cation exchange capacity
- The ability to chemically condition leachate through sacrificial action
- Construction from inexpensive materials
- Tolerance of deformation during service without barrier failure through brittle cracking
- The ability to promote self-sealing of cracks
- Ease of construction
- Sufficient strength to support a refuse vehicle during operation. A cube strength of 5 N/mm^2 is adequate. (The strength requirement is only for emplacement i.e. sufficient for vehicular access. After two years, little strength is required).

The design concept of the new composite landfill liners is to emplace a number of different layers, each of which compliments and enhances the behaviour of the others. Each of the layers has different properties, so that any defects such as cracks, are likely to form at different locations in different layers, thus limiting the creation of connected pathways through the barrier. In the design considered in this work, three layers are envisaged as illustrated in figure 1. The clay-based hydraulic barrier is sandwiched between two layers of concrete.

The function of the top cementitious layer is:

1) To provide a hard wearing surface to protect the lower layers from the effects of vehicular access, waste compaction and damage from sharp objects in the waste.
2) To provide tensile strength in the barrier in order to resist damage from settlement during waste emplacement.
3) To provide an initial barrier to leachate migration (enhanced by the crack sealing properties of the layer below).

The first two requirements are only necessary during the operational phase of the landfill. As this phase is relatively short, this layer will only be required to maintain its structural integrity for a short time. This offers the possibility of using waste materials such as metallurgical slags and spent foundry sands as concrete aggregates in this layer. Although neither material is suitable for use as an aggregate in structural concrete, owing either to slow reactivity with cement or poor bond strength with cement paste, both are suitable for this low-grade application. Co-disposal of demolition and metallurgical

waste will suppress transition metal ion solubility by release of alkalinity from the cementitious binder, whilst offering an opportunity to re-cycle both waste streams with a genuine economic benefit. Currently, both these waste materials are disposed to landfill in Europe and the USA.

The action of the middle clay layer is:

1) To greatly reduce the rate of mass transfer through the barrier by inherently low permeability and high cation exchange capacity.
2) To flow into fissures in the cementitious layers above and below, thus obviating the need for high quality, structural concrete.

Bentonite is not used for this layer because of its high cost and its loss of swelling properties in an alkaline environment. Unlike the swelling clays, kandites and illites are plentiful, relatively inexpensive and could be used in combination with concrete.

The function of the lower cementitious layer is as follows:

1) To provide a blinding layer giving a firm smooth base for the emplacement of the softer material in the middle layer.
2) To provide a final chemical barrier to leachate migration by chemically condition solutions permeating through the liner.
3) To provide a final physical barrier to leachate migration and promote crack healing through secondary mineralisation

The barrier will locally provide a highly alkaline chemical environment, capable of precipitating many metals as relatively insoluble salts, should they start to migrate through it. This will be achieved through sacrificial dissolution of cement hydrates, as leachate slowly percolates through the connective pore network. The chemical performance of the barrier is similar to the backfill grouts used for nuclear waste isolation that was developed on behalf of UK NIREX Ltd. [18]. The chemical barrier will continue to operate long after the physical properties have been lost.

3. Characteristics of secondary by-product / waste minerals used

The various waste or by-product materials used in the laboratory investigation, are listed below:

a) Sodium sulphate slag (non-ferrous metal refining waste - lead industry)
b) Spent borax slag (non-ferrous metal refining waste - silver industry)
c) Ferrosilicate slag (lumps and sand, from non-ferrous metal refining - zinc industry)

d) Ferrosilicate "copper" slag (non-ferrous metal refining waste - copper industry)

e) Soda slag (non-ferrous metal refining waste - lead industry)

f) Chrome Alumina slag (non-ferrous metal refining waste - chromium industry)

g) Cement Kiln Dust, CKD (Calcite-rich from cement manufacture)

h) Run of station ash (Coal fired power generation)

i) Lagoon ash (Coal fired power generation)

j) PFA (Coal fired power generation)

k) Granulated Blast Furnace Slag, GBS (Iron production, un-ground stock)

l) Steel slag (Steel converter slag)

m) Burnt Oil Shale (Bottom ash from oil shale combustion)

n) By-product Gypsum (Titanium dioxide production)

o) Ground Granulated Blast furnace Slag, GGBS (Conventional cementitious slag)

p) Shell foundry sand (Exhausted casting sand, phenolic resin bound)

q) Green foundry sand (Exhausted casting sand bound by natural clay minerals)

r) Sodium sulphate solution (Neutralisation of sulphuric acid with sodium hydroxide)

These waste materials, which were used, can be divided in the three following categories:

- Those materials, which may be used as aggregates in the concrete or mortar layers, such as Spent foundry sands Residues from the castings industry. These materials are principally quartz sands with residues of thermally degraded binders such as clay minerals (green sand) and phenolic resins combined with carbon char (shell sand) and Semi-crystalline slags from the metals refining industry i.e. alkaline sulphates, ferrosilicates and heavy metal–bearing "soda" slags.
- Waste alkalis, which may be suitable activators for cementitious ground, granulated blast furnace slag (GGBS) or Pulverised fuel ash (PFA) i.e. Liquid raffinates such as alkaline sodium sulphate solution produced during acid neutralisation of processing waste.
- Those waste materials which have inherent cementitious properties, like spent borax slag, GGBS and Gypsum 'filter cake' recovered from acid neutralisation arising from pigment manufacture.

The bulk of the materials are wastes from the castings and metals refining industries such as metalliferous slags and spent foundry sands. Laboratory work has focussed on examining these materials as cementitious binders in their own right and as cement replacement materials or as chemical activators for other cementitious materials. This has allowed the solids to be grouped into those

materials which have cementitious properties, those which are relative chemically inert and would be suitable for use as aggregates and to identify any materials which are not suitable for use as liner materials.

Sodium sulphate slags are a product of pyrometallurgical metal refining, and it was thought that they may be leached in order to release residual sodium sulphate to recover an alkaline solution. Unfortunately, this proved not to be the case, the fraction of soluble sodium sulphate remaining in these slags proved too low to be of value. Figure 2 shows the highly porous surface of the sodium sulphate slag. It has a considerable insoluble solids content which is largely siliceous. Initial experiments using this material as an aggregate in a range of different cent types showed that it was slightly reactive towards the cement and dimensionally unstable. The material was considered unsuitable for further study.

One entirely new cementitious material was developed exclusively, the spent borax slag. It is a sodium tetraborate slag containing zinc oxide, finely ground to produce a cementitious matrix. The chemical composition of this material indicates it to have a relatively high resistance to attack by organic acid mixtures, suggesting it to be very suitable for use as a landfill liner material [21].

Soda slags: Non ferrous metal refining relies on the use of fluxes with which minor impurities may be removed from the melt. Pyrometallurgical processes were seen as a potential source of spent sodium carbonate slags, which may contain residual (and hence recoverable) alkalinity. A soda slag from the lead industry has been identified as potentially fitting the requirements of this study as it contains a high residual sodium carbonate content. This material originates in the ISAMELT furnace and contains mixed heavy metals present as both oxides and sulphides in addition to the remaining sodium carbonate.

Figure 3 (left) shows one of the products formed when this material is open to the atmosphere. As the slag is hygroscopic, it absorbs moisture from the atmosphere allowing both dissolution and oxidation of its constituent phases. The crystals seen in the vug are smithsonite (zinc carbonate) and thought to be an artifact of atmospheric aging. Figure 3 (right) shows the complex structure of the slag, containing free lead prills, along with mixed sulphides and carbonates.

Preliminary leaching experiments (figure 4) show the slag to produce highly alkaline (pH >13) and strongly reducing solutions, the E_H being close to the stability limit of water (-0.5 volts).

Aqueous sodium sulphate waste : The secondary lead process recycles a vast quantity of traction batteries, recovering lead, antimony (and minor non ferrous metals alloyed with the lead) polypropylene and other plastics materials, leaving only the spent electrolyte for disposal. This is presently neutralised by reaction with sodium hydroxide to produce a sodium sulphate liquor. Although not fully oxidised to sulphate, this alkaline solution is equivalent to around 11% Na_2SO_4 by mass and therefore is well suited for use as a slag activator. Preliminary experiments showed that reaction with both blast furnace slag and a range of pulverised fuel ashes proceeds rapidly to produce an alkali activated hydration product. This solution was therefore adopted as the most suitable activator for use in this project.

Cement Kiln Dust is a by-product of cement production. It is weakly hydraulic, containing Portland cement minerals along with a considerable quantity of finely divided calcite (figure 5). These particles are collected from the stack by an electrostatic separator and used as a low-grade cementing material. It has been included in this study as a source of relatively inexpensive Portland cement.

Foundry sands are essentially pure quartz sands, with a surface coating of a binder. They are repeatedly recycled; sprue and casting debris being sieved from the sand after each casting operation. After several uses, the adhesive properties of the sand are reduced, as the binder is thermally degraded due to contact with the molten metal. This prevents them adopting the detail of the pattern and at that stage they are disposed of. They are not used as fine aggregates in structural concrete, owing to the presence of residual binder on their surfaces reducing the strength of the paste-aggregate bond. Foundry sands are typically of two types, those with a natural clay binder and those to which a synthetic binder must be added:

Greensand: Strictly, sands with a natural coating of glauconite, but the term is applied to casting sands coated with other clay minerals.

Shell sand: Sands bound by a synthetic binder, such as phenolic resin.

Ferrosilicate slags are ubiquitous products of pyrometallurgy. They are derived from the siliceous gangue minerals present in the ores and are supplied either as chilled granules or fused lump. Both are very hard materials and the energy costs of crushing the fused lumps should be considered if the material is to be adopted for widespread use.

Chrome alumina is a by-product of chromium manufacture and contains approximately 90% Al_2O_3 by mass with Cr_2O_3 and minor magnesium and transition metals. Within the largely glassy matrix are crystalline domains of substituted chromite. The material is supplied as very hard fused lumps.

In optical (figure 7 top), the slag shows repeated a lamellar structure of purple alumina glass alternating with substituted chromite layers. At higher magnification (figure 7 bottom), the chromite layers are seen to comprise many smaller strongly birefringent crystallites of characteristic trapezoidal cross section.

4. Methods

4.1. Screening of materials

The classification of materials (See figure 8) has involved particle size analysis of the solids, optical and electron microscopy, physical testing and both classical and instrumental chemical analysis. From the materials available in this study, it was possible to develop several binder types varying in composition, properties and cost. Figure 9 summarises the binder formulations considered for screening in the experimental programme.

Over one hundred different mixtures with different proportions and materials were prepared and tested for stability in water from the waste samples. Hand-mixed specimens were cured for 4-5 days in 95% relative humidity and 20°C ± 2°C condition and then submerged under 150 ml of water. The pH of the water and physical integrity of the specimens were monitored. Those, which did not disintegrate, were judged satisfactory for further study as potential paste or aggregates phase in the liner mixture when the mix satisfied other chemical and engineering requirements. It was noted that Spent Borax (passing 300μm) could be used as a cementitious material, owing to its ability to set on contact with water [21].

4.2. Laboratory testing

The compressive strength of pastes and mortars were determined by casting 50 mm cubes and the compressive strength of concrete by 100 mm cubes. All the specimens were cured at 95 per cent relative humidity at 20± 2°C and tested according to B.S. 1881,part 116.

The permeabilities of the specimens were determined using a continuous high-pressure flow experiment in which solution is eluted through the materials at pressures up to 10 MPa depending on

the compressive strength of the particular specimen. The apparatus is a modified Hoek Cell [17] and is adapted to measure both the flow and pressure drop across the sample. To maintain the structural integrity of the sample, and prevent flow past its sides, a confining pressure is applied (as in a triaxial cell) around an impermeable sleeve surrounding the sample. By maintaining the pore solution pressure below that of the confining pressure, the internal structure of the barrier material is maintained. In addition to providing a reaction vessel, the cell is used as a constant head permeameter, allowing dynamic measurement of permeability changes over the duration of each experiment. Measurements were made after one sample volume of liquid had passed through the concrete. Assuming an average permeability of 10^{-9} and a maximum leachate head of 1m above the liner (as required in UK landfills) this corresponds to 16 years of exposure in service. Tests were carried out with deionised water and a synthetic (acetogenic) leachate to examine their effects on permeability evolution and buffering capacity of the concrete.

5. Results and discussion

Figures 10 and 11 are the graphs of results from the laboratory work from which the candidate mixes were selected for site trials. The graphs respectively show the water coefficient of permeability and leachate coefficient of permeability against 28-day compressive strength of the mixes studied in respect of the cementitious material used at highest percentage by weight. The selected mixed were intended to have a permeability below 10^{-9} m/s and a strength below 10 MPa. It may be seen that relatively few mixes lie in the required range for both strength and permeability.

Mixes, which have the minimum requirements of the barrier system, are designated within the dotted lines and arrows in Figures 10 and 11. Although higher strength mixes can be still selected from the mixes studied, it is important first, to consider the mixes which have highest negative cost and secondly, to note that with very high strength and / or very low permeability mixes the performance of the barrier may be impaired as they may promote migration of leachate around them and create volumes which do not contribute to the chemical buffering action of the barrier.

6. Conclusions

The results of the investigation into the use of secondary waste minerals as a novel cementitious landfill multi-layer barrier prove the viability of using secondary mineral wastes as component materials in composite landfill liners. The majority of the waste minerals studied satisfy the requirements for a cementitious barrier system. These materials also show good chemical buffering capacity for landfill waste leachate. This work shows that through careful selection and screening, the materials may be combined so as to produce cementitious mixes of low permeability and relatively high resistance to organic acids. These materials have a niche application; they are largely unsuitable for use as structural concrete, yet are very well suited to their roles as barriers to pollution migration.

Concrete or mortar production using waste materials extends the useful life of those materials, which would otherwise form part of the landfill inventory. The commercial and environmental implications of this are far-reaching, disposal costs are negated, transport costs are unchanged and the environmental benefit of re-use is demonstrably great. Moreover, the technical benefits of adopting mineral barrier technology over conventional polymer membranes are attractive.

From these experiments, a number of formulations as barrier mixes were selected to incorporate in field trials. The results of the field trials will be reported when the monitoring of them are completed in an additional paper.

Acknowledgements

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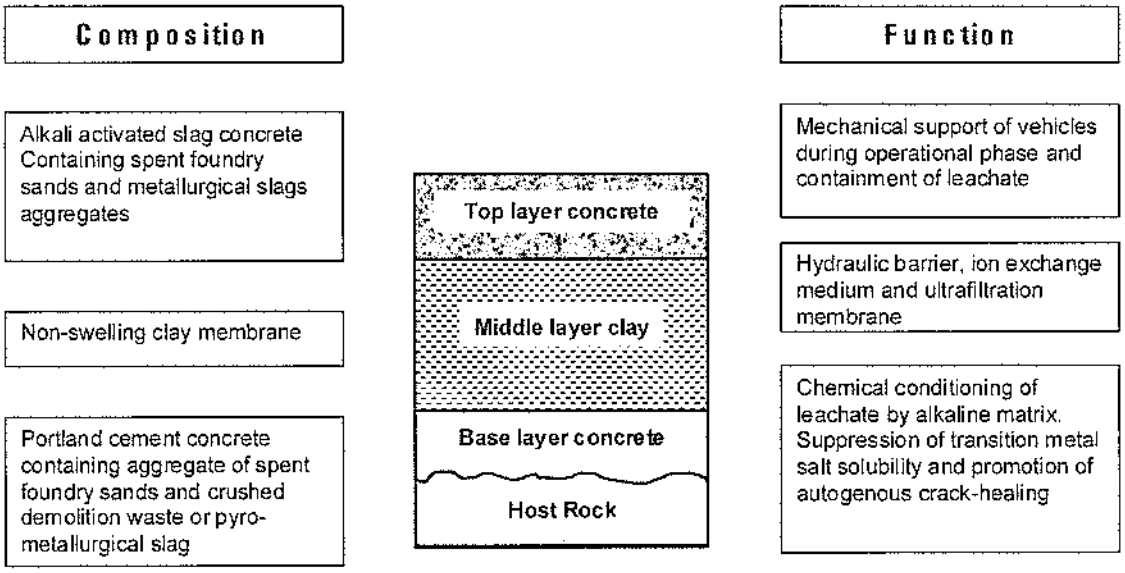

Figure 1: The new composite landfill liner

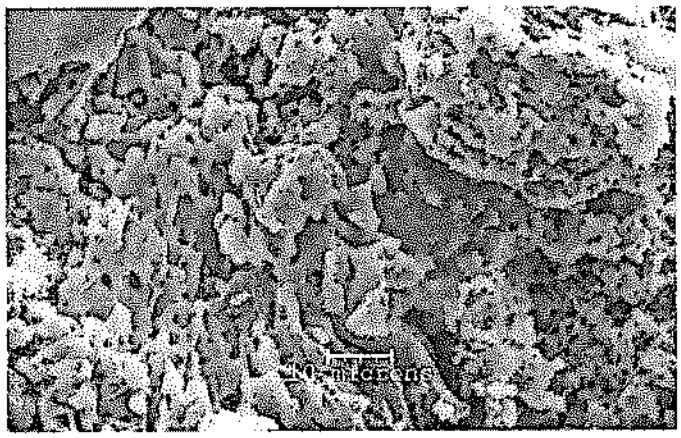

Figure 2: Sodium sulphate slag from the secondary lead industry, Secondary electron SEM image of fracture surface.

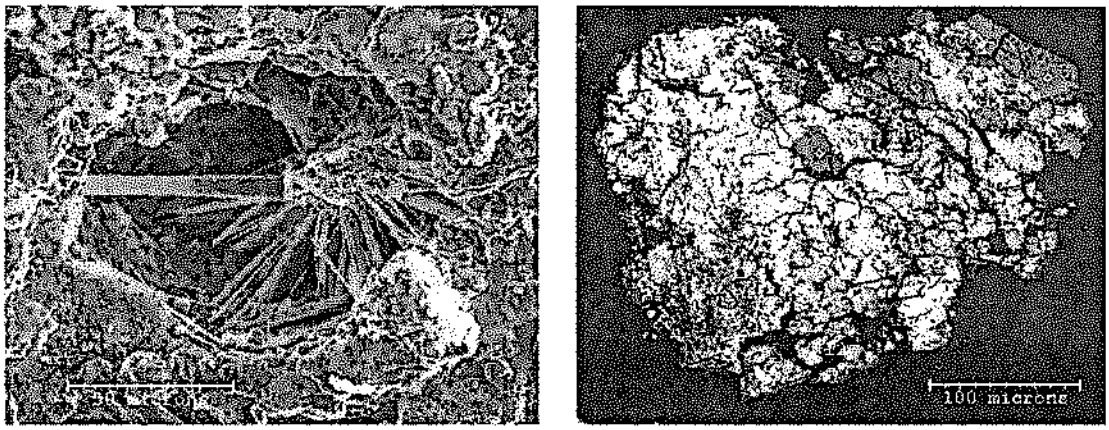

Figure 3: Fresh (right) and aged (left) soda slag prior to leaching.

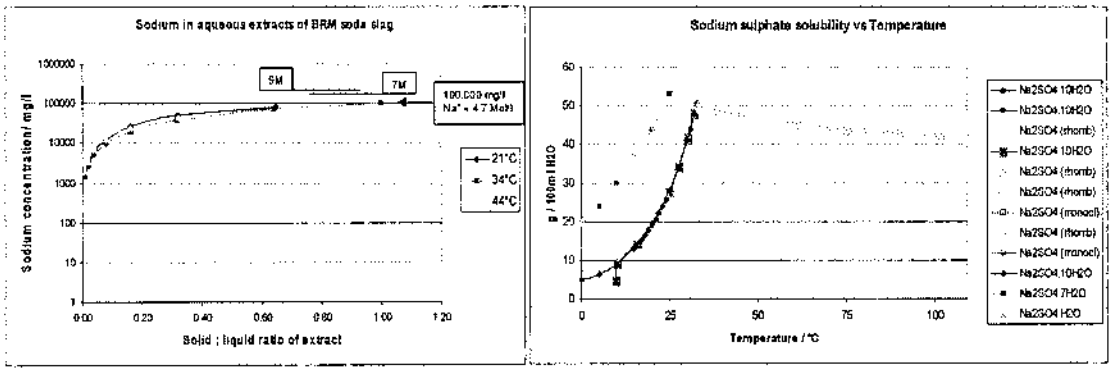

Figure 4: Left: Sodium content of water-leached soda slag as a function of solid:liquid ratio. Right: Thermodynamic solubility of pure sodium sulphate solids (Data from PHRQPITZ).

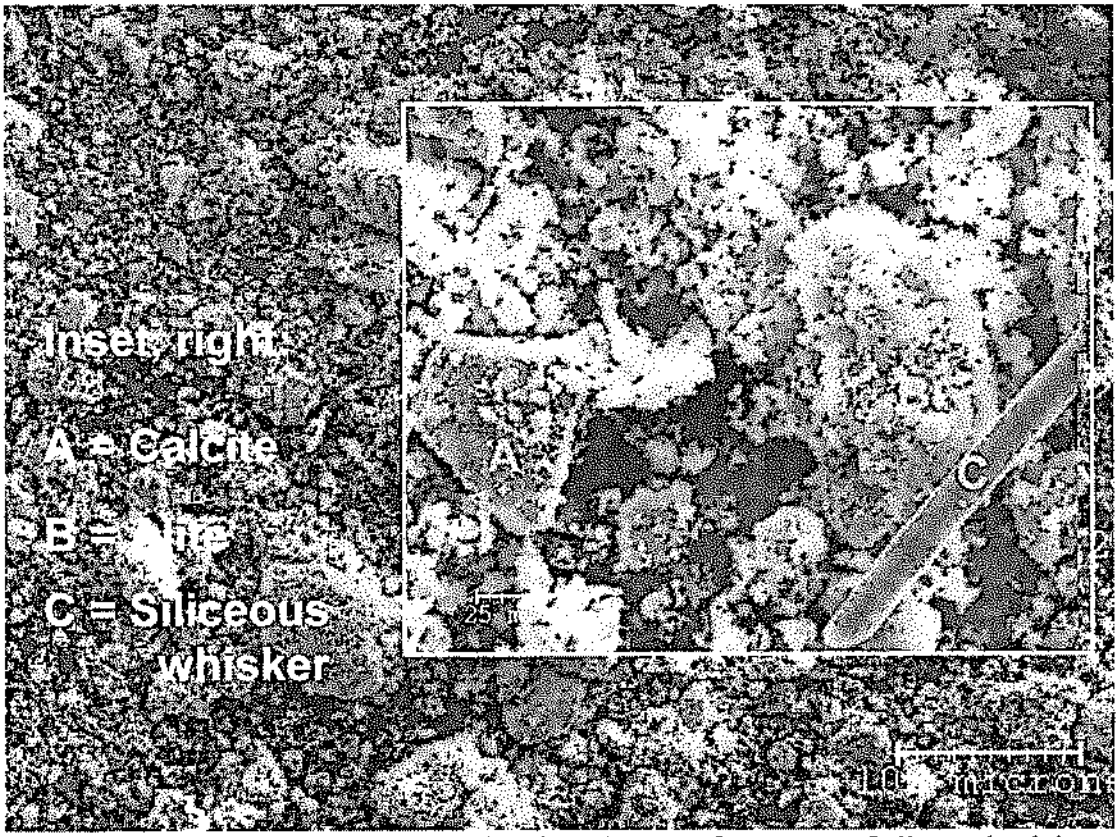

Figure 5: Cement Kiln Dust (CKD) showing cleavage fragments of alite and calcite.

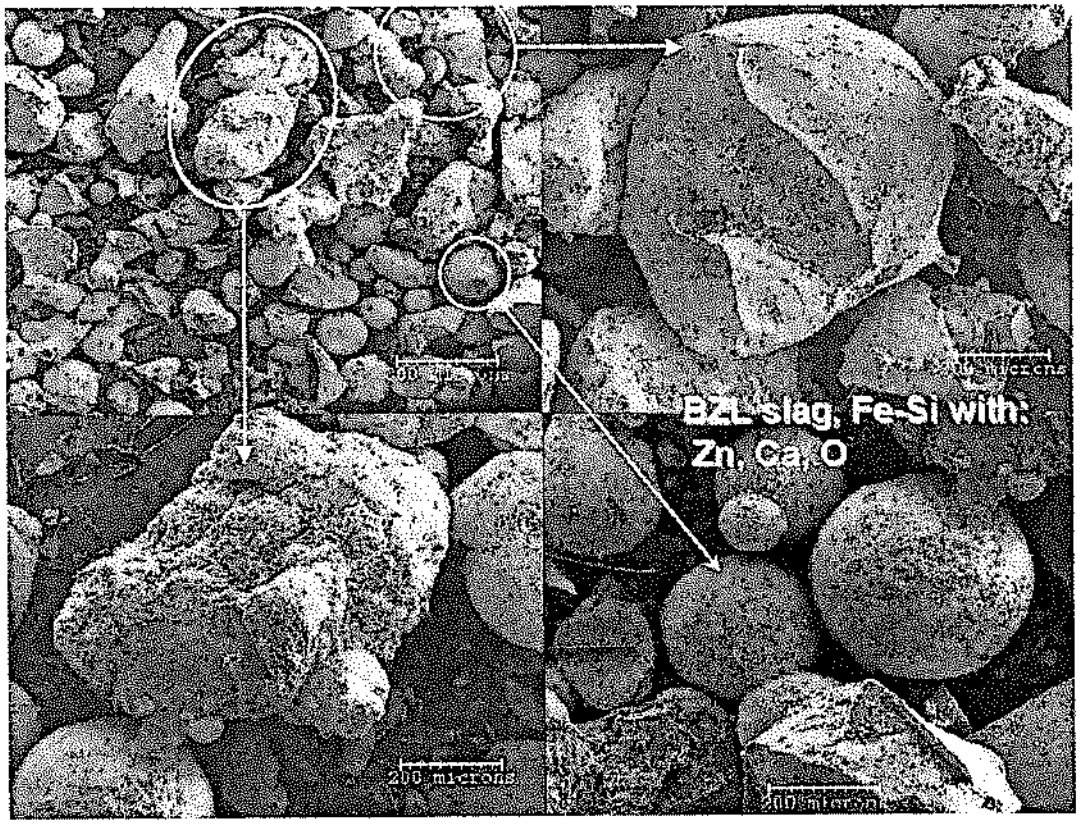

Figure 6: chilled ferrosilicate slag grains produced as a by-product of Zinc refining.

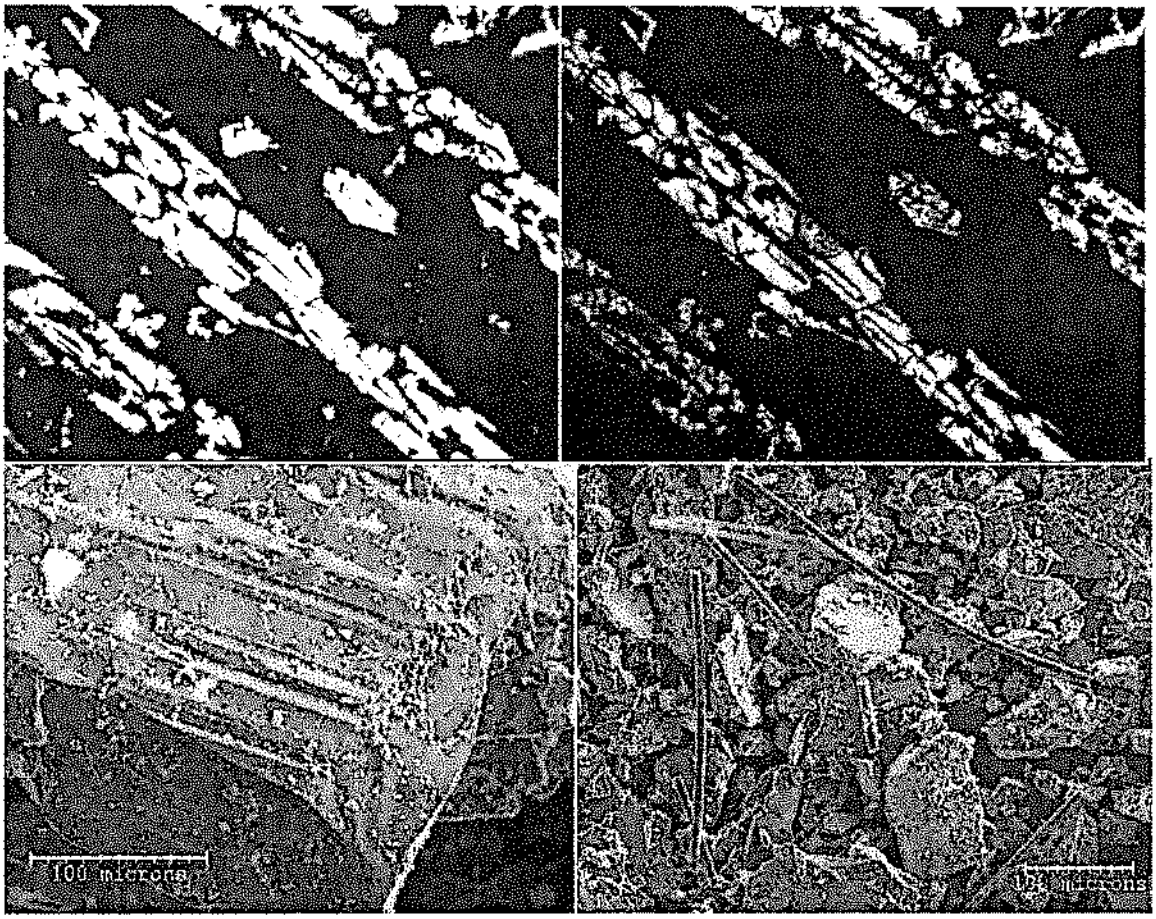

Figure 7: Chrome alumina slag. Above, in optical sections, below, as secondary electron images showing whiskers which appear to be a magnesium silicate.

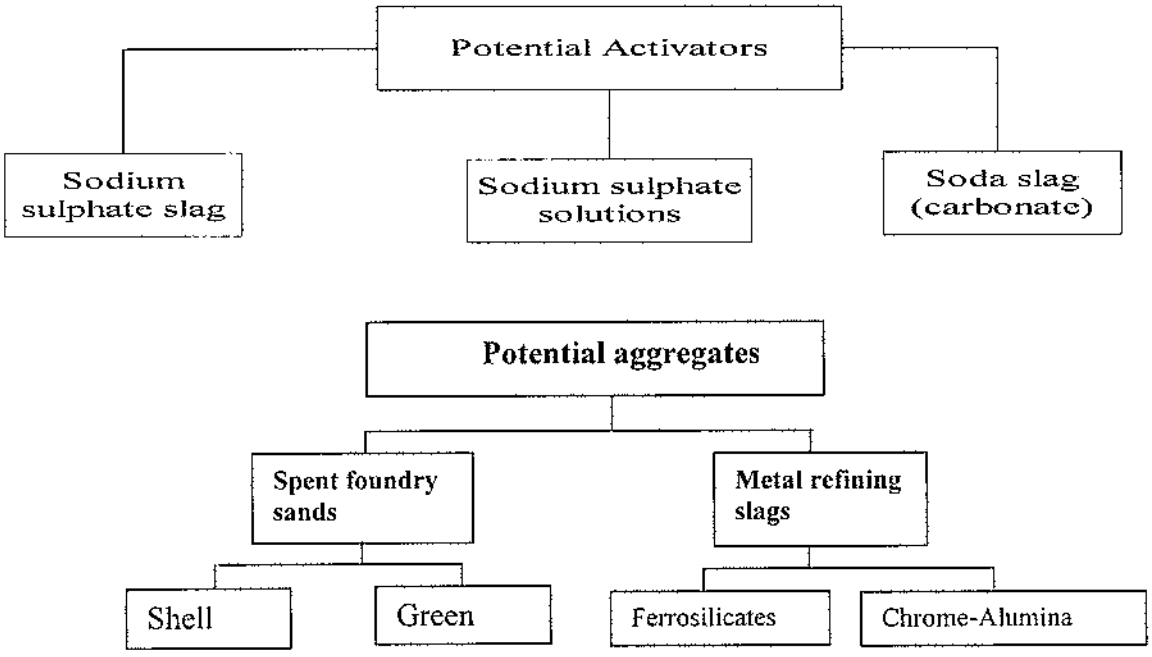

Figure 8: Waste types supplied in the liners project.

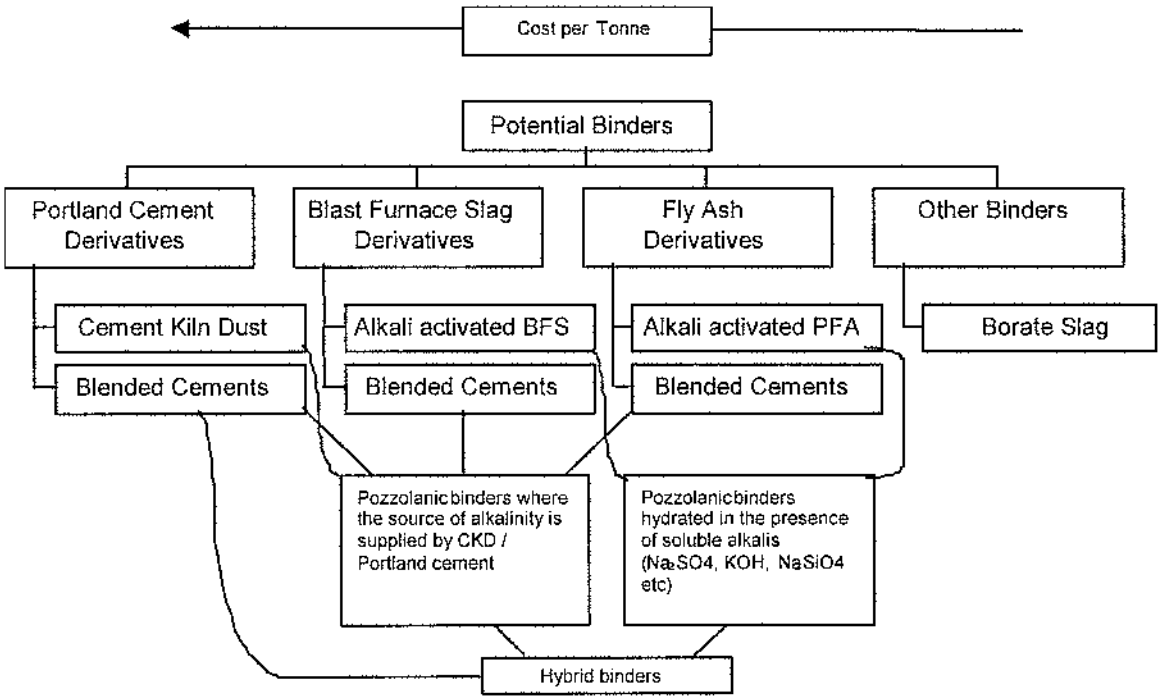

Figure 9: Binder types considered in this study.

Fig. 10: Deionised water coefficient of permeability versus Compressive strength for all mixes.

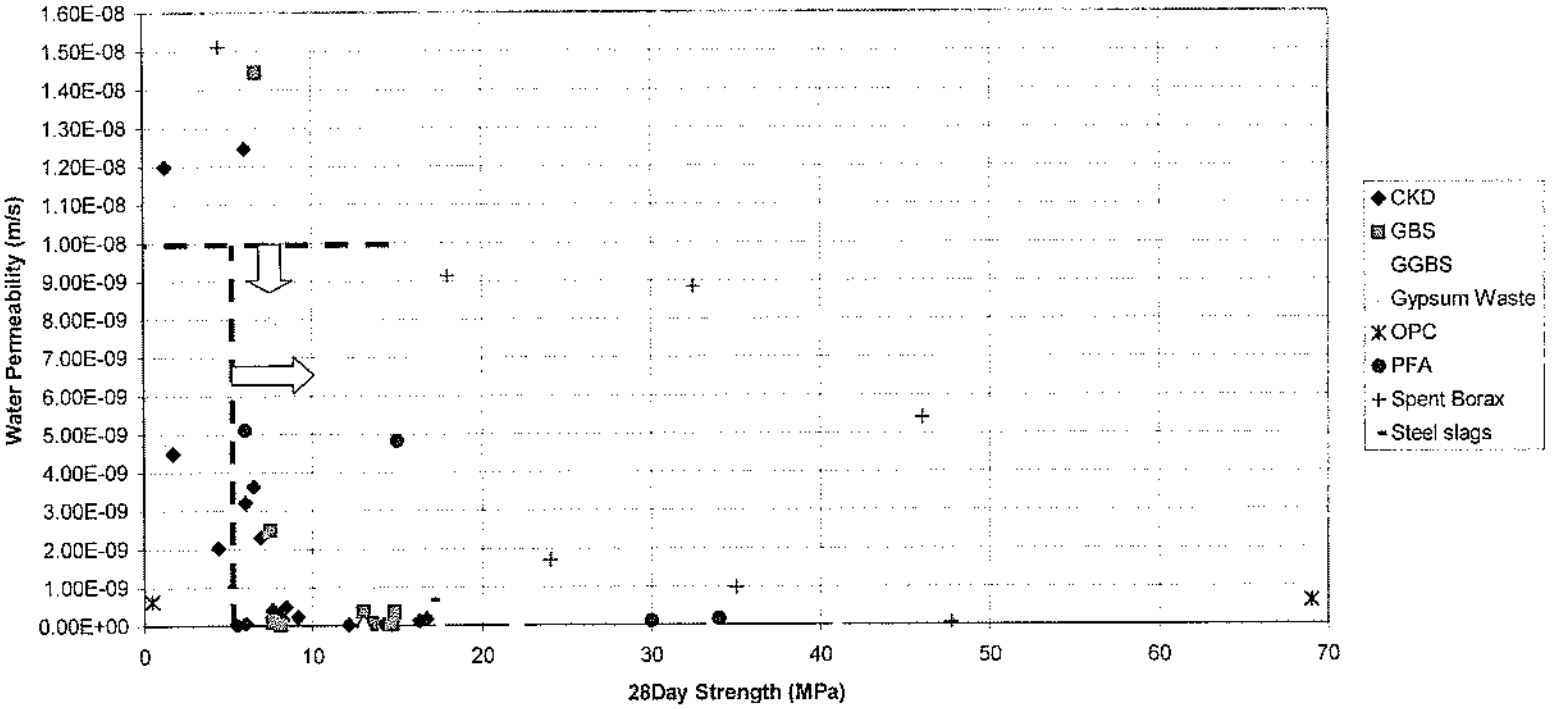

Fig. 11: Synthetic leachate coefficient of permeability versus compressive strength for various mixes.